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Prepared for the
12th International Conference on Composite Materials
cosponsored by the AMAC, CEA, and SF2M
Paris, France, July 5–9, 1999

National Aeronautics and
Space Administration

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Acknowledgments

We wish to thank Mr. T. Kacik for the TGA analyses.

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Summary

In order to better understand SiC fiber behavior within CMC microstructures, mechanical tests were performed on multifilament tows consisting of different types of as-produced and pretreated fibers. Tensile strengths of tows and single fibers were measured at room temperature for nonstoichiometric Hi-Nicalon and ZMI fibers and for stoichiometric Hi-Nicalon-S, Tyranno SA, and Sylramic fibers. Based on simple bundle theory, measured strengths for as-produced and sized tows were in general agreement with the single fiber results. However, after sizing removal under inert conditions, tow strengths for the coarser grained stoichiometric fibers were typically lower than those predicted from individual fiber data. This effect is attributed to enhanced fiber-fiber mechanical interaction caused by sizing removal from the rough surfaces of these fibers. In support of this, tow strengths remained high for those fiber types with fine grains or excess surface carbon; and, when re-coated with a BN interphase coating, tow strengths for the coarser grained fibers returned to their as-produced values. When the tows were pretreated in air at intermediate temperatures, tow strengths decreased in a manner that could be correlated with the oxidation characteristics of each fiber type as measured by thermogravimetric analysis. The creep and rupture properties of Hi-Nicalon and Sylramic tows were also measured in air and argon from 1200 to 1400 °C. Although displaying transient and environmental effects similar to single fibers, the tows crept faster at short times and slower at long times. This resulted in the tow rupture strengths at long time being much greater than the rupture strengths of single fibers. The CMC implications of the tow results are discussed, as well as the benefits and limitations of tow testing.

INTRODUCTION

Continuous-length silicon carbide (SiC) fibers are being developed as reinforcement of ceramic matrix composites (CMC) for high temperature structural applications. Small diameter SiC fibers (~10 to 14 μm) produced by polymer pyrolysis with little or no oxygen content display high stiffness, high room temperature strength, and high thermal stability in comparison to other types of ceramic fibers. Recent examples of these low-oxygen types are the Hi-Nicalon fiber from Nippon Carbon (microcrystalline β SiC with ~5 nm grain size plus excess carbon and trace oxygen) and the Sylramic fiber from Dow Corning (stoichiometric crystalline β -SiC with ~100 nm grain size plus a trace of boron as a sintering additive). They have displayed improved creep and rupture properties compared to the first-generation oxygen-containing Nicalon SiC fiber (refs. 1 and 2). However, at temperatures above ~1300 °C, the nonstoichiometric Hi-Nicalon fiber displays weight loss, grain growth, and contraction; thermal instability effects not observed in the stoichiometric Sylramic fibers (refs. 3 to 4). More recently, Nippon Carbon and Ube Industries have introduced their own stoichiometric SiC fibers, namely Hi-Nicalon Type S (ref. 5) and Tyranno SA (ref. 6), respectively, which have grain sizes and tensile strengths similar to those of the Sylramic fiber. All the stoichiometric types differ, however, in their production processes and in key details of their chemistries and microstructures.

The properties of the small-diameter SiC fibers as described above are typically measured using single fibers removed from multifilament tows containing ~400 to 800 fibers. However, CMC are almost always constructed using woven or braided tows which then become the structural units that provide the CMC with many of its key mechanical properties. Because of the weaving or braiding processes, the fibers are tightly packed in the tows, with typically over 90 percent of the fibers contacting one or more of its neighbors. Also during CMC fabrication, the sizing materials on the multifilament SiC tows are removed and replaced with thin compliant

interfacial coatings (interphases) that have the purpose of protecting the fibers from detrimental interactions with each other, with the matrix, and with the environment during CMC application. Thus mechanical tests on tightly-packed multifilament tows that have been exposed to simulated CMC conditions should be more useful for understanding SiC fiber performance within CMC microstructures than single fiber tests. These tow tests not only allow the evaluation of many fibers in the structural forms used in CMC, but also permit a better understanding of the various fiber surface and load transfer interactions that may occur during CMC fabrication and use. Possible additional advantages of tow testing are the development of quality control procedures for as-produced tows at the fiber vendor or for interphase-coated tows at the CMC fabricator.

The objectives of this study were (1) to measure the tensile strength and creep-rupture properties of multifilament tows consisting of various nonstoichiometric and stoichiometric SiC fibers, and (2) to compare the multifilament behavior with single fiber data measured under the same conditions in order to assess the benefits and limitations of the tow test for understanding SiC fiber performance in CMC. The tensile strength measurements were made at room temperature on tows and single fibers in their as-produced condition, after being precoated with a BN interphase coating, and after being treated under various time, temperature, and environmental conditions that attempt to simulate typical process and use conditions for CMC. The creep-rupture measurements were made at 1200 and 1400 °C on as-produced tows and single fibers under inert (argon) and oxidizing (air) conditions.

EXPERIMENTAL

Six types of SiC fibers available as continuous-length multifilament tows were examined in this study. These included the nonstoichiometric Hi-Nicalon and ZMI (ref. 6) fibers, and the most recent versions of the stoichiometric Hi-Nicalon Type S, Tyranno SA, and Sylramic fibers. Also studied was a recent type of developmental Sylramic fiber, Syl(1), which was specially processed to reduce boron content in the fiber bulk and to form a thin (~150 nm) crystalline BN layer on the fiber surface (ref. 7). Boron removal from the bulk appears to significantly improve the fiber's creep and rupture resistance without degrading fiber tensile strength. Table I lists nominal properties of all the fiber types as available from the manufacturers and the literature. Except for Hi-Nicalon-S, the stoichiometric fibers are typically coarser grained than the non-stoichiometric types and as such have rougher surfaces. Also, except for Syl(1), each type in the as-produced condition was precoated by the manufacturer with a certain type and quantity of polymer-based sizing material. The sizing material typically burns off in argon or air at temperatures from ~200 to 500 °C so that sizing was not present for mechanical tests made on fibers and tows exposed to higher temperatures.

To determine tensile strength, single fibers and tows of all fiber types were tested to fracture at room temperature using a 25 mm gauge length and a constant displacement rate of 1.27 mm/min. The average cross-sectional area for the multifilament tows was assumed to be equal to the filament-count times the average fiber area based on the average diameter for each type. For each tow specimen, the fiber tow was aligned tightly on paper tabs separated by a 25 or 225 mm grip to grip length for room-temperature strength tests or for high-temperature creep-rupture tests, respectively. Detailed procedures for the tow specimen preparation are reported elsewhere (ref. 8). To better understand the effects of environment on the mechanical property data, thermogravimetric analysis (TGA) measurements were also made on the different fiber types in dry air from room temperature to 1500 °C. The TGA specimens consisted of tows wrapped into small volumes with initial weights from ~30 to 50 mg.

The creep-rupture properties were determined for Hi-Nicalon and Sylramic tows using the same fiber creep-rupture facilities and procedures previously reported (ref. 9). Creep deformation versus time was recorded at a constant deadweight load using either a 25 or 100 mm hot zone length for the air furnace and a 115 mm hot zone length for the argon furnace. The creep-rupture properties for Hi-Nicalon and Sylramic single fibers as well as for single fibers of the other stoichiometric fibers have been reported elsewhere (refs. 7 and 10).

Results and Discussion

The room-temperature tensile strengths are shown in figure 1 for (a) single fibers and (b) tows in their as-produced condition and after exposure at 1000 °C for 3 hr in vacuum or at 1400 to 1600 °C for 1 hr in argon. These exposure conditions were chosen to simulate those typical of CMC interphase and matrix formation, respectively. The strength values at each condition were the average of at least 12 single fibers or at least 3 multifilament tows. Standard deviations are also shown in figure 1.

In general, the strength results for the as-produced single fibers were somewhat different than the nominal strength values quoted by the manufacturer for each fiber type (see table I). This is probably due to fiber strength variation from spool to spool. Nevertheless, for the fibers of this study, the average tensile strengths of the Sylramic, Syl(1), and ZMI fibers were the highest, above 2700 MPa; followed by the Hi-Nicalon fiber; while those of the SA and Hi-Nicalon-S fibers were below 1900 MPa, particularly the SA fiber which showed a low tensile strength of ~1400 MPa. It should be noted that this as-produced strength for SA is in qualitative agreement with the large grain size of this fiber in comparison to the other fibers (see table I). After inert exposure in vacuum or argon, the average strengths of single fibers from all fiber types except Syl(1) decreased somewhat. This strength degradation under inert conditions is difficult to understand, particularly for the stoichiometric fibers, which were fabricated at much higher temperatures than the exposure temperatures. It may be possible that at intermediate temperatures from 1000 to 1400 °C, the fiber surface morphologies and chemistries were altered by the exposure environments, giving rise to surface flaws larger than the as-produced flaws for some of the tested fibers (ref. 11). Thus strength retention behavior for any fiber may be strongly dependent on the type and content of impurities on its surface and on the chemical composition of the exposure environment. The strength retention suggests that the crystalline BN on its surface can perhaps protect this fiber from environmental attack.

The strengths of as-produced and inert-treated tows, shown in figure 1(b), were calculated by dividing the tow fracture load by the total cross-sectional area of the bundle. As with single fibers, tow strengths also decreased after inert exposures; but the relative changes were generally greater than that observed for single fibers. In addition, the strength rankings for the various fiber types before and after treatment were somewhat different for tows than for single fibers. To better understand the source of these effects, it was assumed that the fibers in a tow fractured independently so that tow strengths could be calculated using the single fiber results and simple bundle theory (ref. 13):

$$\sigma_b / \sigma_f = (1 / (m e))^{(1/m)} / \Gamma((m + 1) / m) \quad (1)$$

Here σ_b is the predicted tow strength, σ_f is the average measured strength for the single fibers, e is the natural logarithm base, Γ is the gamma function, and m is the Weibull modulus that characterizes the strength distribution of the single fibers. For the as-produced and 1400 °C inert treatment conditions, table II shows the measured average strength and calculated Weibull modulus for the single fibers, the measured average tow strength, and the tow strength predicted by equation (1).

For the as-produced condition, table II shows that measured and predicted tow strengths were in general agreement, except for the unsized Syl(1) fiber and for the weak SA fiber. In general, the observation of tow strengths lower than expected from simple bundle theory suggests that the fibers within these tows did not fracture independently, but that fracture of the weaker fibers caused premature fracture of the stronger fibers. That this effect did not occur in the sized fibers but in the unsized Syl(1) fiber suggests that the sizing materials can aid in reducing detrimental fiber-fiber mechanical interaction within tows, particularly for fibers with rough surfaces. On the other hand, the low strength result for the SA tow suggests that too much sizing (see table I) may also cause undesirable fiber-fiber interaction if the fibers are inherently weak.

After 1 hr exposure in argon at 1400 °C, table II shows that agreement between measured and predicted tow strengths still existed for the Hi-Nicalon types, but now both Sylramic types were weaker than expected. The behavior for both Hi-Nicalon types suggests that even after sizing burn off in argon, sufficient compliance still existed between fibers. This may be due to the fact that these types have smoother surfaces than the coarser grained stoichiometric fibers. Or, they may have retained some intrinsic carbon on their surfaces that acted as a compliant layer between fibers. On the other hand, sizing removal from the coarse grained Sylramic fiber probably enhanced fiber-fiber interaction as in the case of the as-produced and unsized Syl(1) fiber. The important role of surface compliant layers, especially for fibers with rough surfaces, is supported by the fact that the strength of Sylramic tows, although degrading after a 1400 °C inert exposure, actually increased after a 1600 °C exposure (see fig. 1(b)). This is probably attributable to the growth of a compliant intrinsic layer of carbon on the fiber surfaces caused by silicon evaporation from the SiC. Also as shown, when the sizing on a Sylramic tow was thermally removed and then replaced by a compliant, chemically vapor-deposited, BN layer, the tow strength returned to a value equal to its as-produced strength. Thus, unless interphase coatings are applied that are compliant enough to minimize or completely eliminate fiber-fiber mechanical interaction, the potential exists that CMC will display lower strengths than predicted from single fiber data and composite theory (ref. 13). This may not be the case, however, for those fiber types that have fine grains or form an intrinsic carbon layer on their surfaces during either fiber or CMC fabrication.

After air exposure for 100 hr at 400 to 1000 °C, room-temperature tow strengths decreased as shown in figure 2 for (a) the stoichiometric SiC fibers and (b) the nonstoichiometric fibers. The air exposures were cho-

sen to simulate CMC cracking conditions in which the tows may be exposed to oxygen at intermediate temperatures for long times. Typically the tow strengths for all fibers started to drop at $\sim 200^\circ\text{C}$, and then leveled off at ~ 50 to 70 percent of their as-produced strengths at $\sim 600^\circ\text{C}$. The two primary exceptions were the Hi-Nicalon tows which showed a sharp drop in strength of ~ 80 percent from ~ 500 to 600°C , and the Sylramic tows whose strengths remained relatively high and even showed a small increase to more than 1000 MPa near $\sim 900^\circ\text{C}$. However, near 1000°C , the strengths of all tows dropped to about the same low level of $\sim 400\text{ MPa}$. Generally the fracture modes for these low strength tows were characterized by (1) localized failure with many groups of fibers bonded together and (2) one or more fracture planes depending upon degree of fiber-fiber bonding.

To better understand these oxidative effects, the TGA studies shown in figure 3 were obtained on SiC fiber tows in dry air from room temperature to 1500°C at a warm-up rate of $\sim 5^\circ\text{C}/\text{min}$. The SA, Hi-Nicalon-S, ZMI, and Hi-Nicalon tows showed abrupt weight decreases at $\sim 250^\circ\text{C}$, but the Sylramic fiber showed only a small decrease up to $\sim 450^\circ\text{C}$. These results can be attributed to the weight of sizing material on the various fibers (see table I); that is, on a relative basis, less sizing for Sylramic and more sizing for the other fibers. From ~ 450 to 1000°C , the Sylramic fiber showed a weight gain up to ~ 1.3 percent; however, all other types showed only ~ 0.2 to 0.5 percent increase. The weight gain for the Sylramic fiber at intermediate temperatures is probably due to the presence of boron and TiB_2 on the fiber surface (ref. 7). That is, it is well known that liquid boron, which melts at 450°C , can rapidly enhance silica growth on SiC materials (ref. 14). The developmental Syl(1) fiber with reduced boron and a crystalline BN surface appears to measurably reduce this intermediate temperature effect. Above 1000°C , all SiC fibers tested showed a continuous weight increase related to their rates of oxidation. The enhanced oxidation rates for the Sylramic and Syl(1) fibers can be attributed to the boron containing second phases that exist on the surfaces and in the bulk of these fibers.

Returning to the tow results of figure 2, it would appear from the TGA data that the sharp strength drop for Hi-Nicalon from ~ 500 to 600°C was probably due to oxidative removal of sizing and perhaps intrinsic carbon on the fiber surface. In support of the existence of two types of carbon-containing materials, figure 3 shows two low-temperature weight loss stages for both the Hi-Nicalon and Hi-Nicalon-S fibers. The intrinsic carbon on the Hi-Nicalon fiber may not only have allowed compliant interaction between fibers, but also may have healed or covered up large as-produced flaws on the fiber surface. Thus the large strength loss for Hi-Nicalon could be related to the oxidative removal of a variety of beneficial carbon-related effects. For the other fiber types, which degrade slowly in a continuous manner up to $\sim 600^\circ\text{C}$, perhaps only the compliant effects of sizings and surface carbons were lost in air. On the other hand, the tow strength increase near 900°C for the Sylramic fiber appears to be due to the formation of a borosilicate glass surface layer which, if thin enough, can heal surface flaws without chemically bonding the fibers together. Above 1000°C , all fibers began to rapidly form silica on their surfaces, which completely bonded the fibers. This gave rise to tow strengths of $\sim 400\text{ MPa}$, which may represent the minimum strength to be retained by a SiC fiber tow when all the fibers are well bonded by oxidation at intermediate temperatures. For CMC, the oxidation results clearly point to the need for some fiber separation within woven tows, plus minimal contamination with oxygen during the interphase coating process or during composite application.

Typical creep curves for Hi-Nicalon and Sylramic tows and single fibers are shown in figure 4 as measured in argon at 1400°C . Similar time effects were also observed in air. The environmental conditions were chosen to simulate fiber behavior, respectively, in (1) an uncracked CMC and (2) a cracked CMC in which the fibers are bridging matrix cracks exposed to an oxidizing environment. Below $\sim 80\text{ MPa}$, the Hi-Nicalon fibers and tows displayed axial contraction which can be related to further densification of this fiber at the test temperature (ref. 4). Above $\sim 80\text{ MPa}$, the Hi-Nicalon fiber as well single fibers of the other SiC types displayed a transient creep stage, even up to fiber rupture (ref. 10). As with single fibers, the transient creep behavior was more pronounced for the Hi-Nicalon tows than for the Sylramic tows. In figure 4, the rupture strain of the Hi-Nicalon tow was over 1 percent, while that of the Sylramic tow was much <1 percent. In relation to single fibers, the tows generally crept more at short times and less at long times. The short-time effect may be real or an artifact related to some amount of nonuniform loading of the multiple fibers that eventually disappears. The long-time effect could be due to load shifting between fibers of different creep rates or perhaps to some tow-related mechanism that improves the creep resistance of the individual fibers. Clearly more work is needed in this area to understand the sources and CMC significance of the creep rate differences between tows and single fibers. This could be done either experimentally on tows (other fiber types, hot grips, short creep lengths, etc.) or theoretically by analyzing the creep-rupture of mini- and macro-CMC containing Hi-Nicalon or Sylramic fibers.

The effects of stress on the 10 hr -creep strain at 1400°C for the Hi-Nicalon and Sylramic single fibers and tows are shown in figure 5 under (a) argon and (b) air test conditions. In both argon and air, the data for the single fibers follow a power dependence of ~ 2 (ref. 10). In air, the tows follow the same stress dependence, but follow a higher dependence in argon, probably because of lower creep strain at the lower stresses. As

previously discussed, the tows in air are well bonded, so the tow creep data effectively represents the behavior of a unidirectional mini-composite with a high fiber volume fraction. The very good absolute agreement between tow and single fiber creep behavior in air (fig. 5(b)) is probably a consequence of the fact that these measurements were made at 10 hr. That is, at shorter times the fibers crept less than the tows, and at longer times the fibers crept more.

Plots of applied stress (or rupture strength) versus average rupture time are shown in figure 6 for Hi-Nicalon and Sylramic tows and single fibers at 1400 °C in (a) argon and (b) air. In general, the time rate of rupture strength degradation for the tows was much weaker under both environments than that for the single fibers. At short times (or high stresses), the Hi-Nicalon and Sylramic tows displayed lower rupture strengths (or shorter rupture times) than the single fibers. At long times (or low stresses), the tows displayed higher rupture strengths (or longer rupture times) than the single fibers. This general trend, which also occurred at 1200 °C, appears to be related to the difference in instantaneous creep rates of the tows and fibers as described above. Thus understanding the sources of the tow and fiber creep behavior should also shed light on their rupture time behavior. Also shown in figure 6(b) is the rupture strength versus time results for single Syl(1) fibers. This behavior represents the best rupture-resistance observed to date for a single fiber of any small-diameter SiC fiber in air (ref. 7).

Between 1 and 100 hr at 1400 °C in argon, single Hi-Nicalon and Sylramic fibers displayed average rupture strains of ~1.2 and ~0.2 percent, respectively. In air, the rupture strain of the Sylramic fiber, as well as the other stoichiometric fibers, increased by about a factor of two. Apparently in air, oxide formation blunted strength controlling flaws on their rough fiber surfaces; while in argon, flaw blunting by silica formation was not available. For Sylramic tows, the average rupture strain was ~0.5 percent and changed little between air and argon. For Hi-Nicalon tows, the average rupture strain was actually higher in argon (~3.5 percent) than in air (~2.0 percent). Thus, although the rupture strains of the individual fibers in a tow were probably enhanced in air, strong fiber-fiber bonding allowed the weakest fiber to rupture the entire tow at a strain equal to or less than the tow strain in argon.

Summary and Conclusions

In this study, the thermomechanical properties of various nonstoichiometric and stoichiometric SiC tows were evaluated under inert and oxidizing conditions. Tow testing was selected primarily to determine its advantages and disadvantages over single fiber tests in evaluating SiC fiber performance in CMC microstructures.

In regard to tensile strength, it was observed that in their as-produced and sized condition, tows of all fiber types displayed strengths that could be predicted by bundle theory in which it is assumed that all fibers fracture independently. However, after inert thermal treatments that removed the sizing, tow strengths for the coarser grained fibers were typically less than predicted from single fiber data. The lower strengths appear to be related to premature tow fracture caused by fiber-fiber mechanical interaction. This interaction appears to be enhanced by the removal of compliant surface sizings, by the absence of intrinsic carbon surface layers, and by the rough surfaces of the coarser grained fibers. After thermal treatment in air, tow strengths decreased further in a manner dependent on the surface chemistries and morphologies of the SiC fiber types. TGA studies suggest that the primary strength degradation mechanisms were sizing removal, intrinsic carbon layer removal, and fiber-fiber bonding by surface oxide growth.

In regard to creep behavior from 1200 to 1400 °C, both nonstoichiometric and stoichiometric SiC tows displayed transient and environmental effects similar to those observed for single fibers. However, for the same applied stress and test environment, the tows crept faster than single fibers at short times, and slower at long times. Since the tow and single fibers displayed about the same rupture strain, the tow rupture strengths were smaller at short times and greater at long times. Because the underlying mechanism for this difference in creep-rupture behavior is unknown, it is currently unclear whether single fiber or tow testing is more accurate for understanding fiber creep-rupture behavior in CMC.

In conclusion, the results of this study show that the mechanical testing of multifilament tows in combination with single fiber testing can shed important light on the behavior of SiC fiber reinforcement within CMC microstructures. This is the case because the tow tests, besides evaluating the net behavior of multiple individual fibers, can also assess fiber surface interactions with each other and with interphase coatings. These effects can be especially important for woven CMC where tightly packed tows are used to construct the reinforcement architecture. Thus the tow strength results suggest a very strong need to apply and maintain compliant interphase coatings on the newer coarser grained stoichiometric SiC fibers. It may be important that the interphase material be uniformly coated around the entire fiber circumference so that fiber-fiber mechanical

interaction is completely eliminated. On the other hand, the tow creep-rupture results suggest that, when SiC fibers share time-dependent loads with each other, fiber deformation and fracture at high temperatures may be significantly different than that observed with single fibers. Further studies are clearly needed in this area because of its importance not only for understanding CMC thermomechanical performance at high temperatures but also for selecting the proper fiber test method for predicting this performance.

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TABLE I.—NOMINAL PROPERTIES OF SIC FIBERS

| Trade Name | Hi-Nicalon | ZMI | Hi-Nicalon S | SA | Sylramic | Syl. (1) (Devel.) |
|---|-----------------|----------------------|--------------------------|--------------------------|--------------------------|--------------------------------------|
| Manufacturer | Nippon carbon | Ube industries | Nippon carbon | Ube Industries | Dow Corning | Dow Corning |
| Process, (Sintering > 1600 °C) | Polymer-derived | Polymer-derived | Polymer-D./ Sintering | Polymer-D./ Sintering | Polymer-D./ Sintering | Polymer-D./ Sintering |
| Average grain size, ^a nm | 5(XRD) | ----- | 22 (XRD) | > 150 | ~ 100 | ~ 100 |
| Average Surface Roughness (RMS), ^c nm | ~5 | ----- | ----- | ~10 | ~10 | ~ 27 |
| Second phases or impurity conc. (wt.%) | 0.5 O, 36 C | 8.3 O, 35 C, 2 Zr | O(0.2) | Al(<2), O(0.3) | TiB ₂ , B | Reduced B, TiB ₂ , B-N |
| Density, g/cm ³ | 2.7 | 2.48 | 3.0 | 3.1 | 3.1 | > 3.1 |
| Average diameter, μm | 14 | 11 | 13 | 10 | 10 | 10 |
| Number of filaments | 500 | 400 | 500 | 800 | 800 | 800 |
| PVA sizing, wt.% | >1 | (~1.0) ^b | 1.2 | (~1.0) ^b | ~0.2 | - |
| Modulus at RT (GPa) | 270 | 200 | 400-420 | 375 | ~400 | > 400 |
| Tensile strength, ^d MPa | 2800 | 2700 | ~2500 | 2800 | 3200 | ~3200 |
| Tensile elongation, percent | ~1.0 | 1.7 | ~0.6 | ~0.7 | ~0.8 | <~0.8 |

^a Measured on TEM microstructures.^b Peo sizing.^c References 15 and 16.^d As provided by manufacturers.

TABLE II.—STRENGTH CHARACTERISTICS OF SIC SINGLE FIBERS AND TOWS

| Fiber type | Average fiber strength, MPa | As-produced condition, m | Average tow strength, MPa | Predicted tow strength ^a MPa |
|---------------------------------|-----------------------------|--------------------------|---------------------------|---|
| Hi-Nicalon | 2240 | 6 | 1960 | 1520 |
| ZMI | 2700 | 5 | 1770 | 1740 |
| Hi-Nicalon S | 1900 | 3 | 1450 | 1060 |
| SA | 1430 | 3 | 280 | 800 |
| SYLRAMIC | 2800 | 4 | 1550 | 1700 |
| SYL(1) ⁺ | 3060 | 4 | 1270 | 1860 |
| After 1400 °C in Argon for 1 hr | | | | |
| Hi-Nicalon | 1870 | 5 | 1360 | 1210 |
| Hi-Nicalon S | 1490 | 2 | 730 | 700 |
| SYLRAMIC | 2430 | 3 | 1060 | 1350 |
| SYL(1) | 3060 | 4 | 1200 | 1860 |

^a Predicted by bundle theory, eq. (1), + no sizing.

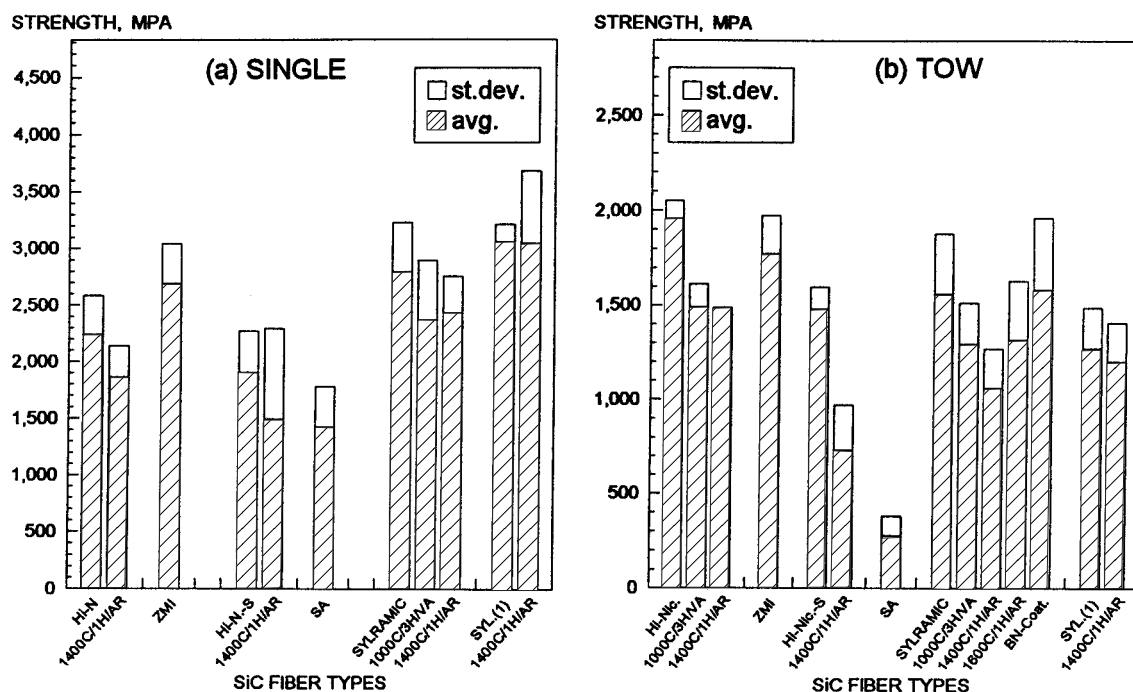


Figure 1.—Room temperature tensile strength of SiC fibers: as-received and after exposure in inert environments for (a) single fiber and (b) tow.

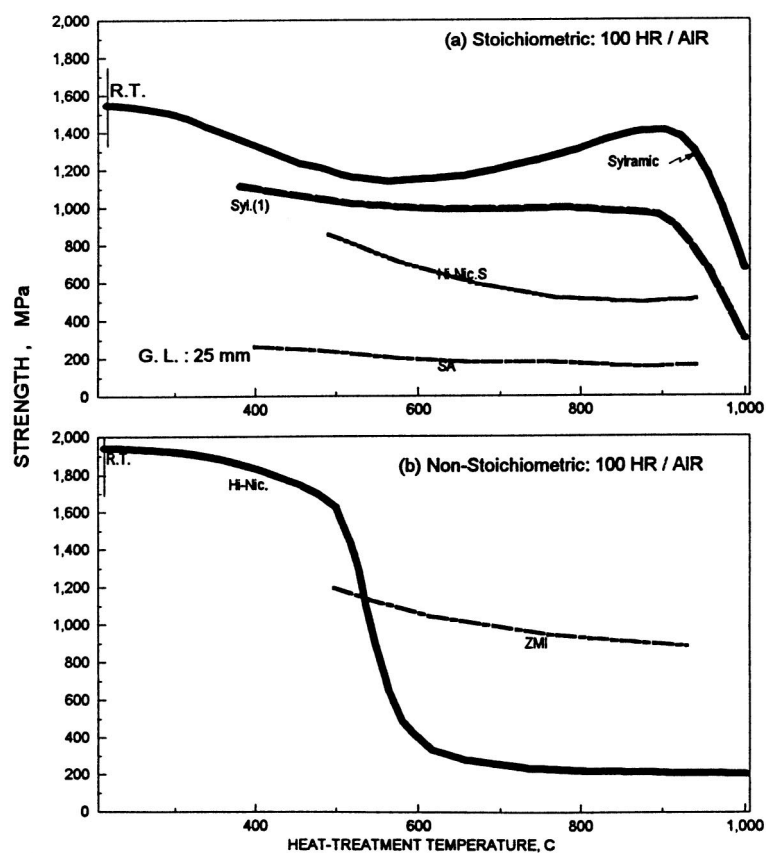


Figure 2.—Room temperature tensile strength after 100 hr air oxidation for (a) stoichiometric and (b) non-stoichiometric SiC tows.

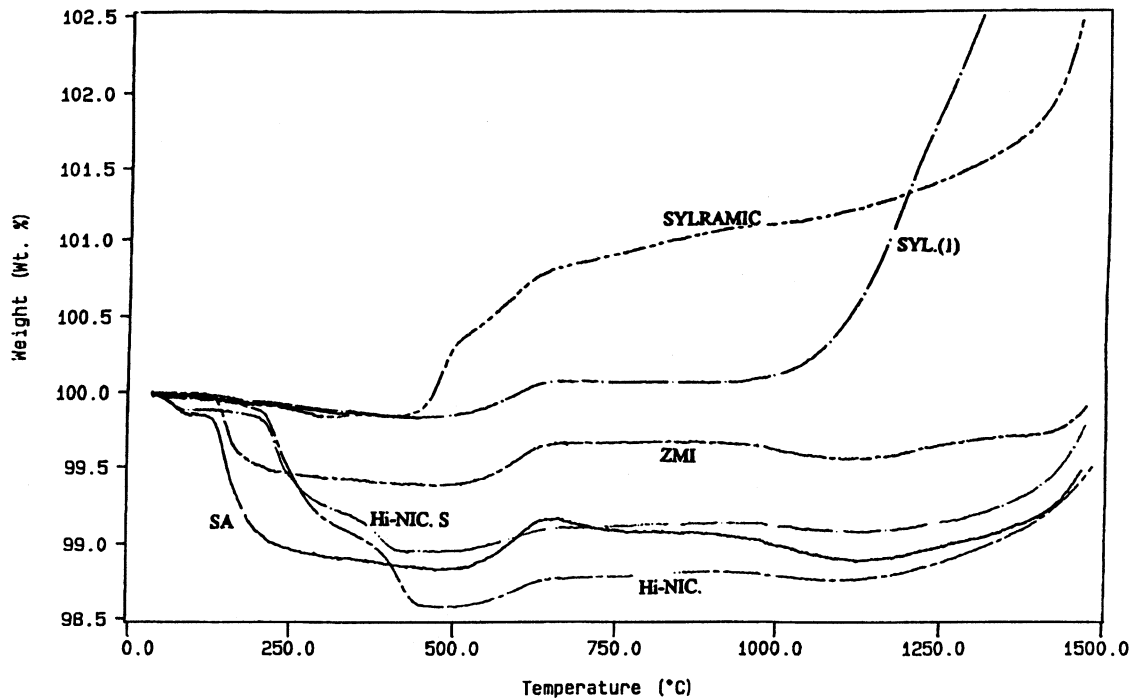


Figure 3.—TGA weight change of as-produced SiC fibers in dry air. Warm-up rate ~ 5 °C/min.

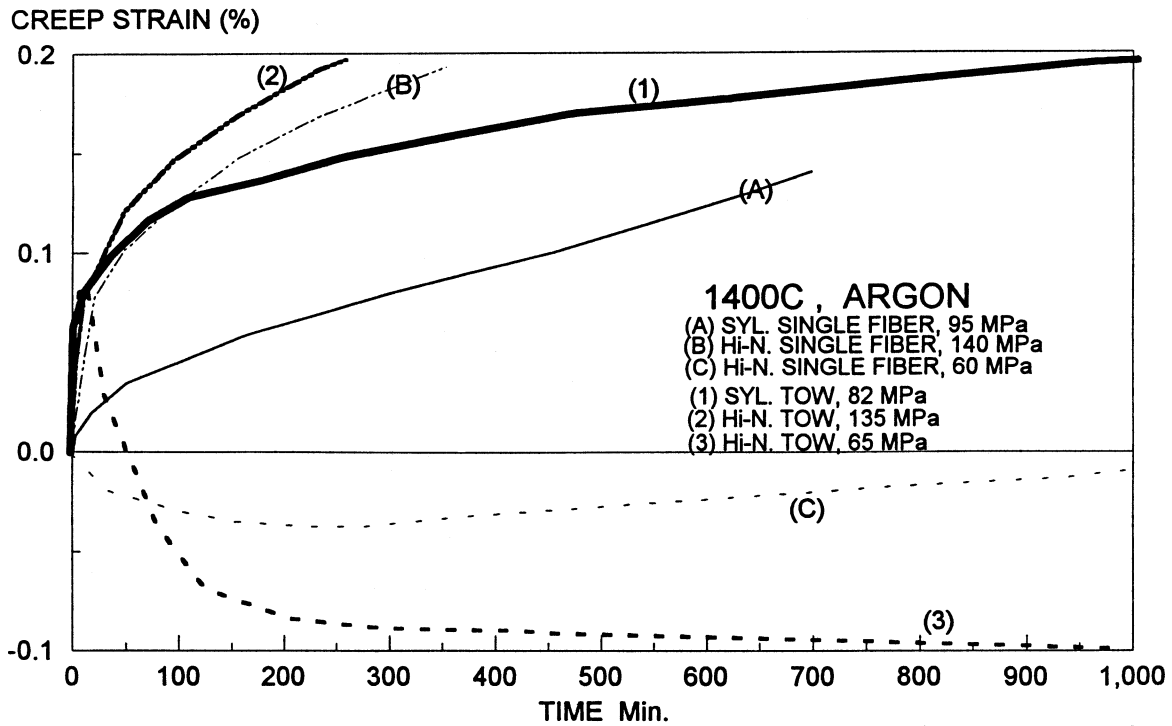


Figure 4.—Typical creep-rupture curves of Sylramic and Hi-Nicalon single and tow fibers at 1400 °C in argon.

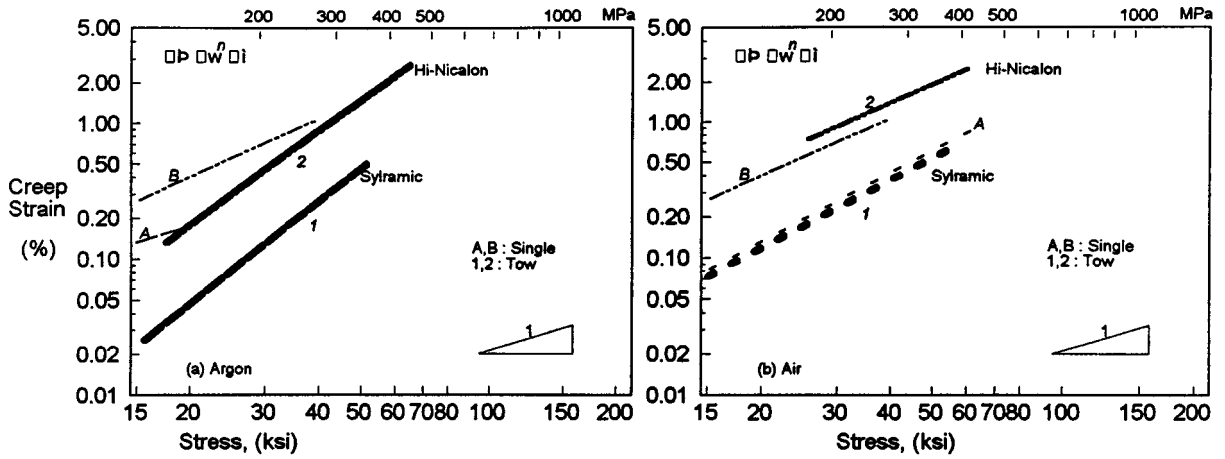


Figure 5.—Stress-effects on creep strain of Sylramic and Hi-Nicalon single and tow fibers after 10 hours at 1400 °C in (a) argon and (b) air.

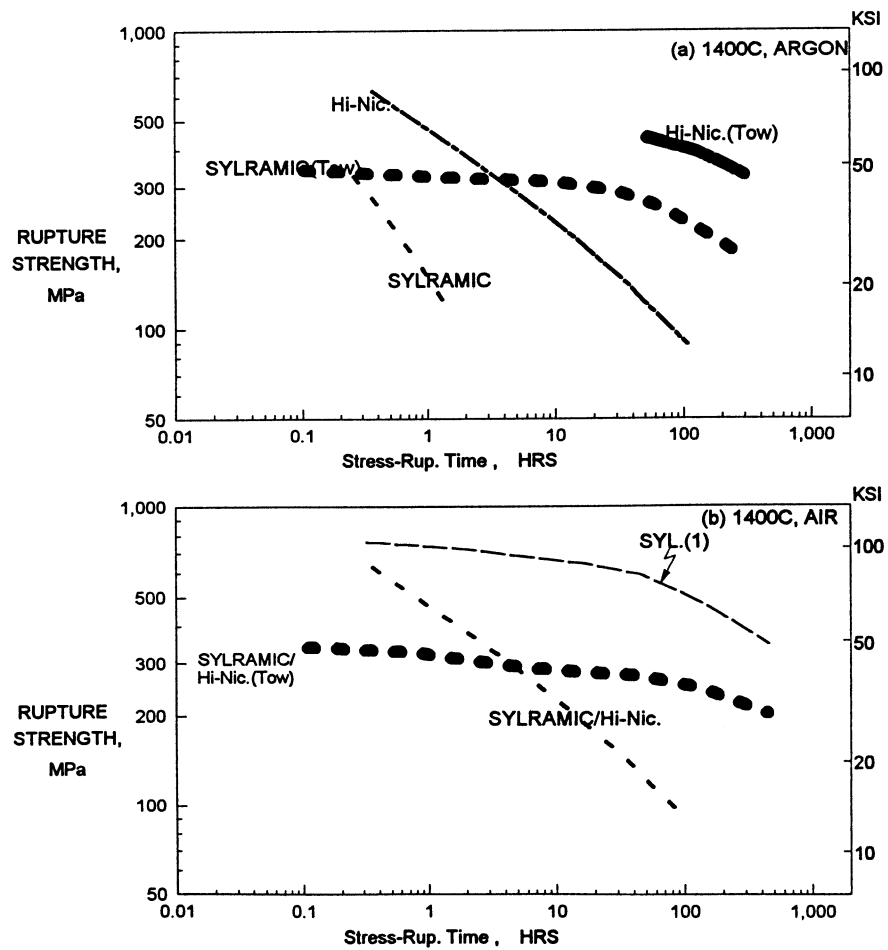


Figure 6.—Rupture strengths of Sylramic and Hi-Nicalon single and tow fibers at 1400 °C in (a) argon and (b) air.

| REPORT DOCUMENTATION PAGE | | | Form Approved OMB No. 0704-0188 | |
|---|--|---|---|--|
| Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503. | | | | |
| 1. AGENCY USE ONLY (Leave blank) | | 2. REPORT DATE July 1999 | | 3. REPORT TYPE AND DATES COVERED Technical Memorandum |
| 4. TITLE AND SUBTITLE Thermomechanical Characterization of SiC Fiber Tows and Implications for CMC | | | 5. FUNDING NUMBERS WU-537-04-20-00 | |
| 6. AUTHOR(S) H.M. Yun and J.A. DiCarlo | | | | |
| 7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration John H. Glenn Research Center at Lewis Field Cleveland, Ohio 44135-3191 | | | 8. PERFORMING ORGANIZATION REPORT NUMBER E-11752 | |
| 9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration Washington, DC 20546-0001 | | | 10. SPONSORING/MONITORING AGENCY REPORT NUMBER NASA TM-1999-209283 | |
| 11. SUPPLEMENTARY NOTES Prepared for the 12th International Conference on Composite Materials cosponsored by the AMAC, CEA, and SF2M, Paris, France, July 5-9, 1999. H.M. Yun, Cleveland State University, Cleveland, Ohio 44115, and J.A. DiCarlo, NASA Glenn Research Center. Responsible person, J.A. DiCarlo, organization code 5130, (216) 433-5514. | | | | |
| 12a. DISTRIBUTION/AVAILABILITY STATEMENT Unclassified - Unlimited Subject Categories: 24 and 27 This publication is available from the NASA Center for AeroSpace Information, (301) 621-0390. | | | 12b. DISTRIBUTION CODE | |
| 13. ABSTRACT (Maximum 200 words) In order to better understand SiC fiber behavior within CMC microstructures, mechanical tests were performed on multifilament tows consisting of different types of as-produced and pretreated fibers. Tensile strengths of tows and single fibers were measured at room temperature for nonstoichiometric Hi-Nicalon and ZMI fibers and for stoichiometric Hi-Nicalon-S, Tyranno SA, and Sylramic fibers. Based on simple bundle theory, measured strengths for as-produced and sized tows were in general agreement with the single fiber results. However, after sizing removal under inert conditions, tow strengths for the coarser grained stoichiometric fibers were typically lower than those predicted from individual fiber data. This effect is attributed to enhanced fiber-fiber mechanical interaction caused by sizing removal from the rough surfaces of these fibers. In support of this, tow strengths remained high for those fiber types with fine grains or excess surface carbon; and, when re-coated with a BN interphase coating, tow strengths for the coarser grained fibers returned to their as-produced values. When the tows were pretreated in air at intermediate temperatures, tow strengths decreased in a manner that could be correlated with the oxidation characteristics of each fiber type as measured by thermogravimetric analysis. The creep and rupture properties of Hi-Nicalon and Sylramic tows were also measured in air and argon from 1200 to 1400 °C. Although displaying transient and environmental effects similar to single fibers, the tows crept faster at short times and slower at long times. This resulted in the tow rupture strengths at long time being much greater than the rupture strengths of single fibers. The CMC implications of the tow results are discussed, as well as the benefits and limitations of tow testing. | | | | |
| 14. SUBJECT TERMS SiC fiber; Tow; CMC; Strength; Sizing; Interphase coating; Oxidation; Creep; Rupture | | | 15. NUMBER OF PAGES 16 | |
| | | | 16. PRICE CODE A03 | |
| 17. SECURITY CLASSIFICATION OF REPORT Unclassified | 18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified | 19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified | 20. LIMITATION OF ABSTRACT | |